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In-depth chemical analysis of electroplating wastewater containing trivalent chromium provides decisive hints for a high efficient membrane based water purification

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Introduction

Analysis of rinse water, electrolyte and RO concentrates

Beside the reduction of metal ions on the product surface, the drag out and rinsing of plated parts is the biggest source of electrolyte loss in the electroplating process. In this way high volumes of wastewaters containing heavy metal ions and other crucial components are created.



1 Scheme of electroplating and subsequent rinsing (blue) and the planned intelWATT membrane-based batch rinse water treatment plant (green), recycling metals and water alike [1]. membrane based process combining water purification and electrolyte recycling is urgently required. A deeper insight into the composition of the wastewater is crucial for the development of an effective treatment.

The ICP-OES analysis of real electroplating rinse water shows high fluctuations in the mass concentrations of Cr(III), SO_4^{2-} , $B(OH)_3$ and TOC, although the concentrations are depicted in relation to the plated and washed area of the parts.

The absolute nickel content, a metal impurity originating from previous plating steps, ranged from 2.58 mg/L to 8.84 mg/L in the rinse water samples. Other metal impurities, like zinc or iron were not detected. The geometry of the plated parts, especially notches and undercuts, cause no simple correlation between the plated area and the contamination of the rinse water.

The organic components of the electrolyte were identified as sodium saccharin and DL-malic acid (see Fig. 3.).



A TOC analysis further confirmed that malic acid is the major source of organic carbon with 6.41 g/L, followed by 1.71 g/L arising from sodium saccharin. The least amount of TOC is caused by the surfactant with 0.03 g/L, due to its low dosing in the electrolyte. The feasibility of reverse osmosis (RO) for the recovery of electrolytic components and water from the rinse waters was to be determined. A diluted Cr(III) electrolyte, containing all organic and inorganic components, was used as feed for the RO trial. The concentrations of the main inorganic mass components in the RO concentrates (K1.1 to K16.1) were determined with ICP-OES (Fig. 4).





of plated and washed parts.

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Fig. 3 ATR-IR analysis of electrolyte components *Replenisher* (identified as sodium saccharin) and Complexor (identified as DL-malic acid).

determined via ICP-OES.

The concentration increase of Cr(III) from 0.74 g/L to 8.39 g/L fits the requirements for re-use in the electrolyte ($\geq 8.0 \text{ g/L}$).

The used membrane (SW30 DuPont) showed promising rejection rates of up to 92.5 % for boric acid and 99.95 % for chromium.

Summary & Outlook

References & Contact

In-depth analysis of real Cr(III) rinse water showed high fluctuations of inorganic components as well as TOC content. The main organic components of the Cr(III) electrolyte were identified as DL-malic acid and sodium saccharin. Nickel was identified as the main metal impurity present in the rinse water.

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Feasibility of RO treatment for rinse water has been proven (99.95% rejection for Cr(III) and 92.5% for boric acid). Resulting RO concentrate fits the concentration for re-use in plating required electrolyte. Future quantification of identified organic components via HPLC should be established.

[1] intelWATT case study 3: Simultaneous metal recovery and wastewater treatment in plastic electroplating production – available at https://www.intelwatt.eu/case-study-3/ (last access 12th Nov 2021)

[2] European Commission, The European Green Deal, European Commission. 53 (2019) 24.

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